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Mechanical Properties of Modified Low Cobalt Powder Metallurgy Udimet 700 Type Alloys

Fredric H. Harf
Lewis Research Center
Cleveland, Ohio

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POWDER METALLURGY UDIMET 700 TYPE ALLOYS

Fredric H. Harf
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

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SUMMARY

Mechanical properties were determined for a series of eight nickel base superalloys derived from low cobalt versions of Udimet 700. The compositions of these alloys had been selected from data provided by a computer program. Material for the alloys was prepared by powder metallurgy and consolidated by hot isostatic pressing. Specimens were given partial solution and aging heat treatments. Tensile tests were performed at room temperature, 650 and 760 °C, and creep rupture tests at 650 and 760 °C. The best properties were developed in an alloy containing in weight percent 8.4 cobalt, 12.9 chromium, 4.85 molybdenum, 4.46 titanium, and 4.36 aluminum, partially solutioned at 1160 °C, followed by a two-step aging treatment at 675 and 790 °C. Its average creep rupture life at 650 °C under 825 MPa stress was 2645 hr. Four more alloys displayed average rupture lives of over 1000 hr, all with low minimum creep rates and adequate ductility. This compares with 252 hr for a powder metallurgy version of Udimet 700. The properties developed by a two-step aging heat treatment exceed those of a four-step aging heat treatment in each of the eight alloys. Alloys with a nominal 8.5 percent cobalt content consistently displayed better creep-rupture properties than those with 4.5 percent or no cobalt, the latter being the weakest.

INTRODUCTION

Cobalt, along with chromium, tantalum and niobium has been designated a "strategic aerospace element," because the United States is almost entirely dependent on imports for the consumption of these elements. The single major use of cobalt is as an alloying element in superalloys, which see service primarily in aircraft gas turbine engines. Reduction of its use is part of a national program named COSAM (Conservation of Strategic Aerospace Materials) (ref. 1). Earlier work has focused on replacing a portion or all of the 17 percent of cobalt in Udimet 700* by the base element nickel and modifying heat treatments to compensate for compositional changes. It was found that, with proper heat treatments, alloys in which up to three fourths of the cobalt had been replaced by nickel had, in the 650 to 760 °C range, properties equaling or exceeding those of the powder metallurgy Udimet 700 alloy (refs. 2 to 4). The objective of the present study was to determine whether changes in other elemental contents of the alloy would further enhance the mechanical properties. Heat treatments appropriate to the composition were to be devised and mechanical properties determined. Should it be possible to develop attractive properties in such modified alloys, they could contribute to the avoidance of future cobalt shortages.

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*Trade name of Special Metals Company.

MATERIALS AND PROCEDURES

Alloys

A series of new alloys, based on Udimet 700, was designed by means of a computer program (ref. 5), which calculates weight fractions, lattice parameters and mismatch of gamma and gamma prime, and propensity toward sigma phase. Of the elements, cobalt contents were restricted to nominally 8.5, 4.5, and 0 percent, the same levels as those used in references 2 and 3. Calculations were performed for about 100 possible alloy compositions with different chromium, molybdenum, titanium, and aluminum contents. From the computed results eight compositions were chosen which had predicted characteristics similar to those of the Udimet 700 and its lower cobalt modifications i.e., gamma prime contents between 40 and 50 wt %, freedom from sigma phase and gamma-gamma prime lattice mismatches of not over 0.15 percent. The compositions of these alloys are listed in table I, along with those of Udimet 700 and lower cobalt variations previously investigated (ref. 3). The alloys were produced by Special Metals Corp. as prealloyed powders. The powders were consolidated by hot isostatic pressing (HIP) in 10 cm long, 18 mm diameter cans for 3 hr at 1215 °C under 140 MPa pressure.

Heat treatment

Since HIP powder metallurgy alloys are suitable for aircraft turbine disks their heat treatment should endow them with optimum properties in the 760 °C and below temperature range. This is usually achieved by partial solutioning of gamma prime plus aging heat treatments. Based on past experience (ref. 3), the temperature for the partial solution was chosen to be 30 to 40 K below the gamma prime solvus, as shown in table II. Specimens from all alloys were partially solutioned in an argon atmosphere for 4 hr and then quenched in oil.

Specimens with two or more different aging treatments were tested of each alloy. These aging heat treatments were to produce a favorable size and quantity of gamma prime particles. As noted in appendix A, it had been observed that the filter clogging by extracted gamma prime varied between alloys and heat treatments and could be measured. In tests of Udimet 700 with reduced cobalt contents the best combinations of properties were obtained when the filter clogging factor (FCF) was about 6000 sec/g. Therefore a preferred choice was that a 6000 sec/g value be represented in at least one of the two aging heat treatments. In its absence a lower value was chosen. One of the heat treatments, also designated as the three-step heat treatment, was selected to have a two-step aging sequence in the range which would produce a uniform size of gamma prime particles. The other heat treatment, designated the five-step heat treatment, would start with two steps in a higher temperature range which would yield fine (100 nm) gamma prime particles to be followed by two aging steps in a lower range for precipitating the remaining solutioned gamma prime as ultrafine (20 nm) particles (ref. 4). Heat treatments given to the alloys, as well as their filter clogging factors are listed in table III. Asterisks indicate heat treatments for which specimens were subjected to mechanical tests.

Mechanical Tests

All mechanical tests were performed in air. Tensile and creep rupture tests were in accordance with ASTM recommended procedures E21 and E139*, respectively. Test specimens are shown in figure 1. The crosshead speed for the tensile tests was 0.5 mm/min. Creep rupture tests used linear variable differential transformers, anchored in grooves machined on the shoulders of the test specimens. Linear extensions were transmitted to a computer for processing into strain and creep rate measurements and data storage.

Metallography

Specimens for metallographic examination were ground and then polished to a 500 nm finish. For optical examination the gamma prime was preferentially dissolved by a solution of 33 percent hydrochloric acid, 33 percent acetic acid, 33 percent water and 1 percent hydrofluoric acid. Specimens for transmission electron microscopy were thinned in a methanol solution with 7 percent perchloric acid and 20 percent butanol. Gamma prime extractions were made electrolytically in an aqueous solution of 1 percent ammonium sulfate and 1 percent citric acid (ref. 6).

RESULTS AND DISCUSSION

Tables IV to VII contain summaries of the tensile and stress rupture test results for the eight alloys. More complete data are contained in appendix B. Where applicable, previously obtained test results of reduced cobalt modifications of Udimet 700 are included for comparison (alloys containing 17, 8.5, and 0 percent cobalt which were the subject of references 3 and 4 are, in this paper, named HE83, HE79 and HE74, respectively).

All the tested alloys displayed superior tensile and stress rupture strengths and lower creep rates with the three-step heat treatment, as compared to the five-step one. Ductilities in general were equivalent within each alloy with either type of heat treatment. Since all the alloys had different compositions, and their heat treatments also varied within the three- and five-step approaches, a direct comparison is difficult to make. However, overall the best combination of properties belongs to alloy HE43, whose superior tensile properties are compared in figure 2 to those of powder metallurgy Udimet 700, also known as Astroloy (ref. 7). In creep rupture at 650 °C and 825 MPa alloy HE43 displays average lives of 2645 and 750 hr with the three- and five-step heat treatments, respectively. The comparable life of alloy HE79, the Udimet 700 modification with an 8.5 percent cobalt content given a five-step heat treatment is 428 hr (ref. 3).

There is a distinct improvement in creep rupture strength, as the cobalt content increases. Thus the shortest rupture lives and highest minimum creep rates were recorded in the cobalt-free alloys HE47 and HE48, while the alloys with 8.5 percent (HE 41, HE42 and HE43) were at the other end of the scale. This is graphically shown in figures 3 and 4 which show the rupture lives of

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*Recommended practice for -E21 elevated temperature tension -E139 conducting creep, creep-rupture, and stress-rupture - tests of metallic materials.

alloy HE43 and compare the average creep rupture lives under three test conditions of all alloys with the same cobalt contents when given the two different types of heat treatments. Such lower strength of the cobalt-free alloys was also observed in the tests of the modifications of Udimet 700 alloy (refs. 2 and 3).

The excellent results obtained by heat treatment K in the 8.5 and 4.5 percent cobalt alloys should also be mentioned. The two aging temperatures of this three-step heat treatment are 25 and 30 K above the last two steps of the "conventional" temperatures used in references 2 to 4, and again here in the two final steps of the five-step heat treatments, as well as in the three-step type E heat treatments. The higher temperatures result in a larger size for the finest gamma prime (ref. 8). This also is indicated by the FCF data for heat treatment types E and K, because the FCF for type K is always less than for type E, due to reduced filter clogging by larger gamma prime particles (table III).

The micrographs in figure 5 taken at 500X of alloy HE43 given the five- and three-step heat treatments are typical for these alloys. The particles visible are partially solutioned gamma prime, measuring about 1000 nm. More typical details are illustrated in the transmission electron micrographs of figure 6, showing the distribution of the fine (100 nm) and ultrafine (20 nm) particles in the microstructure in alloy HE41 with the five-step heat treatment T (fig. 6(a)) and their extracted shapes (fig. 6(b)). The particle sizes are identical to those previously identified in lower cobalt Udimet 700 alloys (refs. 3 and 4). Figure 7, a transmission electron micrograph of the same alloy with the three-step heat treatment K features a nearly uniform particle size (about 50 nm). As expected, the particles for this heat treatment are larger than the ultrafine particles of the five-step heat treatment, because of the temperature increase in the final two aging steps (ref. 8).

CONCLUDING REMARKS

It had been previously established that Udimet 700 did not require 17 percent cobalt for optimum properties, but that 8.5 or even 4.5 percent should suffice (refs. 2 to 4). On the basis of this information a series of alloys was designed with minor variations in other elements, and produced by powder metallurgy. Confirmation was obtained that the alloys in this cobalt range are of high strength. The data indicate, however, that the properties of alloys with an 8.5 percent cobalt content are superior to those with 4.5 percent cobalt, and that the properties are further degraded when cobalt is absent. It was also demonstrated that the properties of Udimet 700 variations with reduced cobalt contents can be enhanced by minor compositional changes. The best combination of properties was produced with alloy HE43, which in a nickel base contains 8.4 cobalt, 12.9 chromium, 4.85 molybdenum, 4.46 titanium, 4.36 aluminum, 0.024 carbon, and 0.017 carbon in weight percent. The preferred heat treatment for this alloy is a partial solutioning of 4 hr at 1160 °C, followed by a two-step aging sequence of 24 hr at 675 °C and 8 hr at 790 °C. Alloy HE43 and several others examined here compare very favorably with the available superalloys, and it is recommended that they be included in any consideration of lower cobalt-content alternatives.

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TABLE I. - ANALYZED COMPOSITIONS OF ALLOYS

Alloy	Co	Cr	Mo	Ti	Al	C	B	Ni
	Weight percent							
HE41	8.5	14.9	5.00	3.54	4.09	0.026	0.018	Bal.
HE42	8.4	12.9	5.25	4.29	4.35	.028	.017	
HE43	8.4	12.9	4.85	4.46	4.36	.024	.017	
HE44	4.5	13.1	4.85	4.34	4.35	.025	.017	
HE45	4.5	11.5	5.80	4.31	4.35	.030	.018	
HE46	4.45	9.9	6.90	4.43	4.35	.024	.018	
HE47	0.0	13.7	5.05	4.35	4.30	.035	.018	
HE48	0.0	11.6	6.25	4.39	4.33	.040	.017	
HE83[a]	17.0	14.8	5.10	3.58	4.04	.06	.026	
HE79[a]	8.5	14.8	5.00	3.54	4.08	.06	.022	
HE74[a]	0.0	15.0	5.00	3.51	4.04	.065	.019	
Astroloy[b]	min 16.0	14.0	4.5	3.35	3.85	.02	.02	
	max 18.0	16.0	5.5	3.65	4.15	.03	.03	

[a]Alloys HE83, HE79, and HE74 are the variations of Udimet 700 with 17, 8.5, and 0 percent cobalt, respectively (ref. 3).
[b]Low carbon version of powder metallurgy Udimet 700, aim composition (ref. 7).

TABLE II. - EQUILIBRIUM[a] AND PARTIAL SOLUTION TEMPERATURES

Alloy	Liquidus, °C	Solidus, °C	Gamma prime solvus, °C	Partial solution temperature, °C
HE41	1343	1250	1165	1130
HE42	1338	1232	1195	1160
HE43	1338	1228	1200	1160
HE44	1337	1250	1210	1171
HE45	1341	1247	1218	1177
HE46	1343	1247	1225	1185
HE47	1333	1242	1215	1171
HE48	1338	1250	1235	1195

[a]Determined by differential thermal analysis.

TABLE III. - HEAT TREATMENTS

[Temperatures in °C.]

Alloy	Type	Partial solution[a]	Aging steps, temperature/hr; air cool				FCF,[b] sec/g
HE41	T**	1130	870/8	980/4	650/24	760/8	6 848
	B*	1130	870/8	1030/1.5	650/24	760/8	9 409
	E	1130	650/24	760/8	------	-----	11 300
	K*	1130	675/24	790/8	------	-----	8 400
HE42	T**	1160	870/8	1010/2	650/24	760/8	2 782
	A**	1160	870/8	1040/1.25	650/24	760/8	5 724
	B*	1160	870/8	1065/1	650/24	760/8	6 167
	D**	1170	925/6	1065/1	650/24	760/8	6 009
	E	1160	650/24	760/8	------	-----	8 230
	K*	1160	675/24	790/8	------	-----	6 061
HE43	T*	1160	870/8	1010/2	650/24	760/8	4 768
	B	1160	870/8	1065/1	650/24	760/8	4 187
	E	1160	650/24	760/8	------	-----	8 710
	K*	1160	675/24	790/8	------	-----	5 182
HE44	T	1171	870/8	1020/1.5	650/24	760/8	3 567
	B*	1171	870/8	1075/1	650/24	760/8	4 146
	E	1171	650/24	760/8	------	-----	7 211
	K*	1171	675/24	790/8	------	-----	5 515
HE45	T*	1177	870/8	1025/1.5	650/24	760/8	3 288
	B	1177	870/8	1085/1	650/24	760/8	3 091
	E*	1177	650/24	760/8	------	-----	6 225
	K	1177	675/24	790/8	------	-----	4 472
HE46	T	1185	870/8	1035/1.5	650/24	760/8	1 926
	B*	1185	870/8	1090/1	650/24	760/8	2 239
	E*	1185	650/24	760/8	------	-----	6 225
	K	1185	675/24	760/8	------	-----	2 375
HE47	T	1171	870/8	1020/1.5	650/24	760/8	2 083
	B*	1171	870/8	1080/1	650/24	760/8	2 922
	E*	1171	650/24	760/8	------	-----	4 316
	K	1171	675/24	790/8	------	-----	2 215
HE48	T	1195	870/8	1045/1.5	650/24	760/8	2 874
	B*	1195	870/8	1100/1	650/24	760/8	3 418
	E	1195	650/24	760/8	------	-----	6 102
	K*	1195	675/24	790/8	------	-----	6 078
HE83[c]	*	1104	870/8	980/4	650/24	760/8	8 398
HE79[c]	*	1130	870/8	980/4	650/24	760/8	6 659
HE74[c]	A[d]*	1145	870/8	980/4	650/24	760/8	3 894
	B[d]*	1145	870/8	1030/2	650/24	760/8	6 841
	G[d]*	1145	870/8	1050/1	650/24	760/8	5 207

[a]Four hours, argon atmosphere, oil quench.
[b]Filter clogging factor (see appendix A).
[c]Alloys HE83, HE79, and HE74 are the variations of Udimet 700 with 17, 8.5, and 0 percent cobalt, respectively (ref. 3).
[d]Heat treatments on cobalt-free alloy (ref. 4).

*Heat treatments subjected to full mechanical test program.
**Heat treatments used for mechanical screening tests (see appendix B).

TABLE IV. - 650 °C TENSILE TEST RESULTS

Alloy	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
Three-step heat treatments					
HE41	K	1361	1057	24.6	25.7
HE42	K	1390	1085	21.5	21.3
HE43	K	1387	1080	28.9	23.0
HE44	K	1337	1043	28.0	22.8
HE45	E	1339	1040	21.0	19.9
HE46	E	1361	1042	13.0	12.1
HE47	E	1369	1070	18.2	17.1
HE48	K	1363	1046	15.9	12.6
Five-step heat treatments					
HE41	B	1255	944	28.9	31.9
HE42	B	1238	948	22.8	24.2
HE43	T	1247	948	29.8	26.5
HE44	B	1175	880	29.8	29.6
HE45	T	1201	909	23.2	22.2
HE46	B	1167	977	9.7	13.5
HE47	B	1174	880	22.6	17.2
HE48	B	1173	882	10.4	16.4
HE83[a]	--	1244	914	15.0	17.6
HE79[a]	--	1227	926	22.6	29.4
HE74[a]	B	1240	900	21.7	13.6

[a]Alloys HE83, HE79, and HE74 are the variations of Udimet 700 with 17, 8.5, and 0 percent cobalt, respectively (ref. 3).

TABLE V. - AVERAGE CREEP RUPTURE TEST RESULTS

[Temperature 650 °C, stress 825 MPa.]

Alloy	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
Three-step heat treatments					
HE41	K	1422	0.25×10^{-8}	6.9	6.0
HE42	K	2088	.21	5.6	3.9
HE43	K	2645	.18	6.6	8.4
HE44	K	1418	.35	5.4	4.4
HE45	E	1036	.26	6.4	2.9
HE46	E	578	.30	4.5	3.9
HE47	E	455	.72	4.5	2.9
HE48	K	281	1.9	7.2	4.5
Five-step heat treatments					
HE41	B	378	1.9×10^{-8}	6.3	4.9
HE42	B	657	.79	6.0	5.6
HE43	T	750	.61	5.9	5.3
HE44	B	391	1.9	9.6	3.4
HE45	T	403	1.0	6.8	6.0
HE46	B	203	2.0	4.7	3.2
HE47	B	176	3.0	5.7	4.8
HE48	B	165	3.0	6.0	4.6
HE83[a]	--	252	1.0	3.2	6.8
HE79[a]	--	428	1.7	5.1	8.3
HE74[a]	B	267	2.7	3.8	14.8

[a]Alloys HE83, HE79, and HE74 are the variations of Udimet 700 with 17, 8.5, and 0 percent cobalt, respectively (ref. 3).

TABLE VI. - CREEP RUPTURE TEST RESULTS

[Temperature 650 °C, stress 900 MPa.]

Alloy	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
Three-step heat treatments					
HE41	K	324	0.9×10^{-8}	4.5	6.2
HE42	K	987	.57	3.4	3.0
HE43	K	683	1.0	5.0	5.0
HE44	K	422	1.4	6.9	4.8
HE45	E	274	.81	2.0	3.2
HE46	E	152	1.3	4.5	2.1
HE47	E	93	2.3	5.9	2.9
HE48	K	137	4.3	4.5	7.5
Five-step heat treatments					
HE41	B	64	9.8x10-8	3.9	6.9
HE42	B	152	5.8	7.4	4.7
HE43	T	267	2.9	5.9	2.3
HE44	B	93	9.6	7.8	4.4
HE45	T	73	7.3	4.9	3.2
HE46	B	90	6.1	4.9	4.2
HE47	B	45	27.0	9.8	8.7
HE48	B	12	----	5.9	1.5

TABLE VII. - CREEP RUPTURE TEST RESULTS

[Temperature 760 °C, stress 400 MPa.]

Alloy	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
Three-step heat treatments					
HE41	K	777	0.36×10^{-8}	3.0	1.4
HE42[a]	K	500	.55	3.2	2.0
HE43	K	596	.50	1.0	3.7
HE44	K	610	.13	0	1.2
HE45	E	327	.30	1.0	3.2
HE46	E	281	.83	1.5	2.3
HE47[a]	E	224	.84	0	1.2
HE48	K	89	4.3	4.5	7.5
Five-step heat treatments					
HE41	B	49	6.4×10^{-8}	2.0	5.4
HE42	B	233	1.1	1.0	1.0
HE43	T	125	2.9	1.4	2.3
HE44	B	108	2.2	.5	.7
HE45	T	98	2.4	.5	1.7
HE46	B	141	1.6	0	2.4
HE47	B	48	4.5	.7	2.1
HE48	B	50	2.9	2.0	2.1

[a]Average.

APPENDIX A

FILTER CLOGGING BY EXTRACTED GAMMA PRIME AND ITS MEASUREMENT

INTRODUCTION

Most nickel-based superalloys contain gamma prime, a Ni_3Al face-centered intermetallic phase, which acts as a strengthening agent. Gamma prime can form from the melt, precipitate from the solid solution matrix at lower temperatures, or do both. It is stable at low temperatures, but at elevated temperatures, all or a portion of it can dissolve in the face-centered matrix of the alloy. Therefore, by heat treating, randomly sized, randomly distributed gamma prime can be removed from the microstructure and reprecipitated in a manner intended to control the properties of an alloy.

The strength of heat-treated superalloys depends largely on the quantity and size distribution of well dispersed gamma prime particles (refs. 3 and 4). The dispersion is controlled by how evenly the alloying elements have diffused throughout the structure during the heat treatment, hence longer times at temperature favor a more even dispersion. The weight fraction of gamma prime in an alloy results from its chemical composition. The quantity of particles depends on their size, which in turn is generated by time and temperature in the aging range, because the particles grow more rapidly at higher temperatures. Heat treatments with more than one aging step may introduce several sizes of gamma prime particles into an alloy, and the proportion of the several sizes can represent the optimum of combined properties for a particular application (refs. 3, 4, and 7). Thus the microstructure of Udimet 700, after a complex heat treatment of partial solution at 1105 °C and four sequential agings at 870, 980, 650, and 760 °C, contains gamma prime particle sizes of about 1000, 100, and 20 nm (refs. 2 and 4), as shown in figure A-1.

The fineness of the smaller particles requires examination by electron microscopy for size measurements and electrolytic extraction for the determination of weight fractions. In extracting gamma prime from Udimet 700 alloy it was observed that the time needed to filter the extract of an approximately equal quantity of particles after the various heat treatments varied widely, depending on the particle size found to be present. This led to the ensuing study.

The author recognizes the contribution made to this work by Sandra Eng, a student from Columbia University, who interned during a summer at the Lewis Research Center.

FILTER CLOGGING MEASUREMENT

Gamma prime is extracted from nickel base superalloys electrolytically in an aqueous solution containing 1 percent ammonium sulfate and 1 percent citric acid (ref. 6). The gamma matrix dissolves in the solution, releasing the gamma prime, which sinks to the bottom of the beaker. This residue is collected and separated from the solution by filtration. From the residue, characteristics such as weight fraction, composition, particle sizes, distribution, shape and lattice parameters can be determined.

In the present work specimens of two different alloys were used. These specimens, of about 14 mm in diameter and 5 mm in thickness, were measured to within 0.1 mm and their surface area calculated. A platinum wire was then welded to each specimen, and the weight of the assembly determined. The specimen constituted the anode, the cathode was a cylindrical platinum screen surrounding the specimen. The electrolyte was contained in a 250 ml glass beaker, and both the anode and the cathode were submerged in it. Power from a dc supply was controlled by current regulation and set at 75 mA/cm^2 of specimen surface. Approximately 100 mg of extracted gamma prime was to be extracted from each specimen.

The extracted gamma prime was separated from the solution by means of the filtering apparatus shown in figure A-2(a), available from Millipore Corporation, Bedford, MA. The apparatus comprised one each

- 300 ml funnel with ground glass seal (fig. A-2(b))
- 47 mm diameter filter
- Ground glass base with coarse glass frit filter (fig. A-2(b)) having a filtration area of approximately 9.6 cm^2
- Aluminum spring clamp (fig. A-2(b))
- Neoprene stopper (fig. A-2(b))
- 1-liter filtering flask
- Vacuum hose
- Vacuum pressure pump

The filter was Durapore* hydrophylic type GVWP, made of polyvynilidene difluoride, with nominal 0.22 μm pore size. Its typical flow rate for water is given as 15 $ml/min/cm^2$ of filtration area at a differential pressure of 7 kPa (520 mm Hg). (The vacuum pump used in the experiment produced 8 kPa (600 mm Hg) differential pressure.)

In preparing the apparatus for the measurements a filter had been preweighed to the nearest 0.01 mg, inserted into the filtering apparatus, and the time determined to vacuum-filter 100 ml of distilled water through it. Then the solution containing the extracted residue was poured into the funnel and the residue captured by the filter. To this was added the residue recovered from the metal specimen by rinsing and ultrasonic cleaning. When the filtering was completed, the vacuum pump was turned off. Exactly 100 ml of distilled water was carefully poured into the funnel, so as not to disturb the collected residue. The pump was then restarted, and the length of time required for the water to pass through the filter with the residue measured. Now the filter with the gamma prime was removed from the apparatus, dried, weighed to the nearest 0.01 mg, and the weight of gamma prime determined by difference. The weight of the residue in gram was used as divisor for the time difference for 100 ml to pass through the filter with and without residue. The value so calculated in seconds per gram is the "filter clogging factor" (FCF) for the specimen. Table A-I is a form with details and sample entries for the procedure. Normally two runs were made for each specimen, and the resulting FCF's tended to fall within 10 percent of the higher value. In the few instances where the difference between FCF's was greater, additional runs were made.

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*Trademark of Millipore Corporation, Bedford, MA.

RESULTS AND DISCUSSION

The results of some of the extractions performed on two variously heat treated alloys are summarized in table A-II. Extracted weight and weight fractions of gamma prime, times to filter 100 ml of water and calculated filter clogging factors are given. In most cases two extraction runs were sufficient to obtain FCF's within the preset 10 percent limits. It was not necessary to perform more than three extractions, weighing between 67 and 110 mg, on any specimen.

The FCF is to a large extent controlled by the pore size of the filter and its thickness. Oversized pores will entrap more of the superfine particles and thereby reduce the flow rate, lengthening the filtering time and raise the FCF. Undersized pores, on the other hand, prevent some of the fine particles from entering the filter, causing them to settle on its surface, where, without support from the fine weave of the filter, they present less of an obstruction to passage of the liquid. A confirmation of this concept is evident from the several extractions which were run for 1 hr on specimens of alloy HE42 and yielded extracts of over 250 mg. It can be assumed, that after the filter pores were filled, the extracted gamma prime remaining on top of the filter offered, on the basis of weight, easier passage to the water, for in every case these FCF's were significantly lower than those of the extractions run for shorter times.

A comparison of the FCF's shows differences between the heat treatments. These distinctions can be correlated with the amounts of ultrafine particles present, as was postulated when this study was initiated. In alloy HE74,[a] as reported in reference 4, the quantity of ultrafine gamma prime increased as the temperature of the second aging step was raised in the four-aging-steps heat treatments A and B. The FCF for heat treatment A is definitely less than for B. In heat treatment G, where noticeable coarsening of the ultrafine gamma prime had been encountered, the FCF was decidedly lower.

Again, in alloy HE42 raising the temperature of the second aging step increased the FCF (heat treatments T, A, and B). The omission of the first two aging steps (heat treatment E) produced considerably more of the filter-clogging, ultrafine particles, which became less numerous and larger when the temperature of the final two aging steps was raised (heat treatment K). Indeed, the FCF for heat treatment K is in the range of those of heat treatments B and D.

CONCLUDING REMARKS

There is strong evidence in heat treatable superalloys for a relation between filter clogging and microstructure. However, the work reported here should be regarded as preliminary, since essentially only one standard was used for the extractions, namely a quantity of near 100 mg. The values of the filter clogging factors from the few extractions performed for one hour and yielding over 250 mg of gamma prime were entirely different. It is left to future investigators to determine what quantity of extracted gamma prime will

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[a]A cobalt-free version of Udimet 700 alloy.

yield results which can distinguish most accurately between different heat treatments. The method could provide an easy way of checking on the accuracy of heat treatment, comparing the response of different heats of an alloy to heat treatment, and otherwise controlling the thermal history of a superalloy. It should be possible to make yet closer correlations between microstructure and filter clogging factors, and furthermore establish a tie-in with alloy properties.

In performing this experiment, some precautions must be taken. The filtering medium, filtering apparatus, and suction must remain identical for determining comparative filter clogging factors, and the weight of gamma prime extracted should not vary greatly between specimens to be compared. Again, additional work is necessary to define the parameters of this procedure. But in its simplicity, it provides a ready means by which, in a short time, one can screen quantities of materials that otherwise might require extensive tests and microstructural investigations.

TABLE A-I. - PROCEDURE, FORM AND SAMPLE ENTRIES

GAMMA PRIME PHASE EXTRACTION

Work sequence	Specimen identity: HE42-D	Run 1	Run 2	Units
1	Preclean specimen, 30 min @ 75 mA/cm^2, triple rinse (before run 1 only)			
2	Specimen area	4.8	4.3	cm^2
3	Specimen weight - original [A]	5.89608	5.67845	g
14	Specimen weight - final [B]	5.72247	5.49232	g
	Specimen weight - loss [W = A - B]	0.17361	0.18613	g
13	Filter and residue, weight [C]	0.21134	0.21624	g
4	Filter, weight [D]	0.11659	0.11389	g
	Residue, weight [R = C - D]	0.09475	0.10235	g
5	Current (specimen area * 75 mA)	360	320	mA
	Extraction time	20	25	min
6	Current, time on	10:00	9:47	clock
9	Current, time off	10:21	10:12	clock
7	Pour about 50 ml H_2O into funnel and filter			
8	Filter 100 ml H_2O, must have 2 runs within 2 sec accuracy [E]	69	69	sec
10	Clean specimen twice for 2 min in H_2O ultrasonically, extract residue by filtration			
11	Filter 100 ml H_2O through filter with residue [F]	633	690	sec
12	Time difference [T = F - E]	564	621	sec
	CALCULATIONS			
	Filter Clogging Factor [T / R]	5952	6067	sec/g
	Gamma prime [R / W]	54.6	55.0	percent

TABLE A-II. - SUMMARY OF GAMMA PRIME EXTRACTIONS

Alloy and heat treatment	Gamma prime, percent	Run number	Weight gamma prime extracted, g	Time to filter, 100 ml H_2O sec	Filter clogging factor, sec/g
HE74-A	47.5	1	0.10708	413	3856
		2	.10276	404	3931
		avg			3894
		3	.10653	482	4525
HE74-B	47.2	1	.06790	461	6789
		2	.10488	723	6893
		avg			6841
HE74-G	47.0	1	.10293	548	5324
		2	.10195	519	5091
		avg			5207
		3	.09684	601	6206
HE42-A	54.2	1	.09422	520	5518
		2	.09272	550	5932
		avg			5724
HE42-B	54.4	1	.09633	594	6166
		2	.10038	621	6187
		avg			6176
		3	.09952	666	6692
HE42-D	54.8	1	.09475	564	5952
		2	.10235	621	6067
		avg			6009
HE42-E	52.5	1	.10093	820	8124
		2	.09656	805	8337
		avg			8230
HE42-K	52.0	1	.10087	602	5968
		2	.09424	580	6154
		avg			6061
HE42-A		*	.26104	722	2766
HE42-B		*	.32239	1176	3648
HE42-D		*	.27700	1178	4253

Notes: Average filter clogging factors were calculated from runs 1 and 2, in which they are within 10 percent of each other. Run 3 exceeds these limits.
*These single extractions were run for 1 hr.

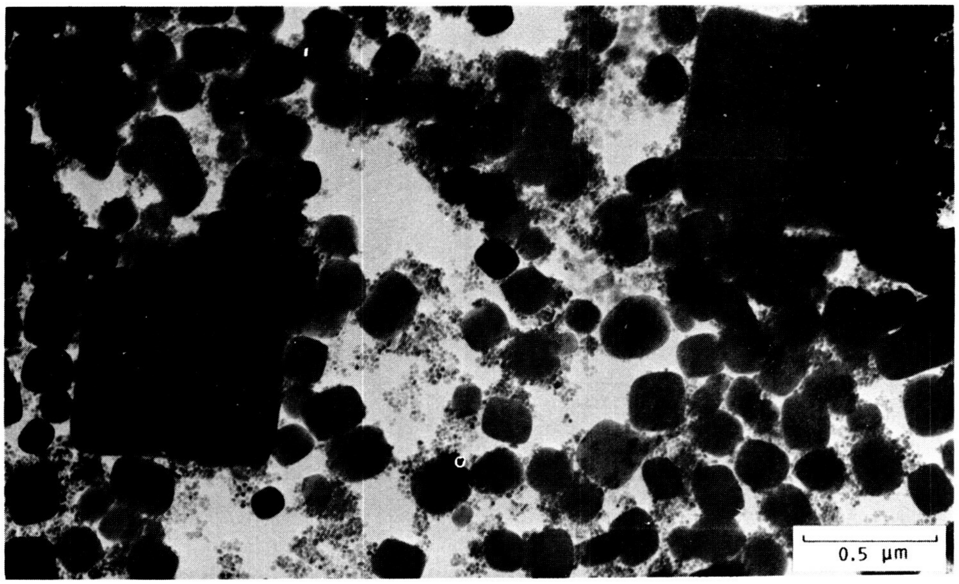

FIGURE A-1. - TRANSMISSION ELECTRON MICROGRAPH OF GAMMA PRIME PARTICLES EXTRACTED FROM HOT ISOSTATICALLY PRESSED POWDER METALLURGY UDIMET 700 ALLOY. THE DISK TYPE HEAT TREATMENT HAS PRODUCED THREE PARTICLES SIZES, MEASURING APPROXIMATELY 1000, 100 AND 20 µm.

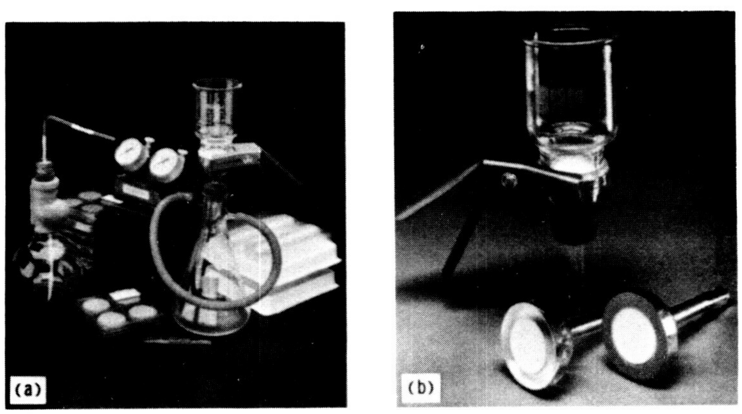

FIGURE A-2. - APPARATUS USED IN PARTICLE EXTRACTION: (a) ASSEMBLY, (b) FUNNEL SUBASSEMBLY AND GLASS FRIT FILTERS.

APPENDIX B

MECHANICAL PROPERTY TEST RESULTS

The following tables contain the results of all the tensile and creep rupture tests run on alloys HE41 through HE48. Also included are average property data obtained in a program designed to determine the effect of lowering the cobalt content in Udimet 700 alloys (refs. 3 and 4).

TABLE B-I. - TENSILE TEST RESULTS FOR ALLOY HE41

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	T	1492	1053	22.5	29.0
	T	1503	1069	22.5	13.5
	B	1486	1042	25.5	24.3
	K	1504	1143	11.5	17.7
650	T	1272	1007	11.4	19.9
	T	1137	979	5.4	12.6
	B	1255	944	28.9	31.9
	K	1361	1057	24.6	25.7
760	B	985	942	6.9	3.5
	K	1031	1014	9.7	11.6

TABLE B-II. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE41

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	T	449	4.9×10^{-9}	7.8	11.6
		T	406	6.3×10^{-9}	6.6	8.8
		B	644	1.4×10^{-8}	6.9	5.9
		B	197	2.6×10^{-8}	7.9	7.4
		B	293	1.6×10^{-8}	4.0	1.3
		K	1557	2.3×10^{-9}	7.9	8.4
		K	1287	2.8×10^{-9}	5.9	3.6
	900	B	64	9.8×10^{-8}	3.9	6.9
		K	324	9.0×10^{-9}	4.5	6.2
760	475	T	16.3	1.7×10^{-7}		
	450	T	18.3	1.4×10^{-7}	5.4	3.5
	400	T	28	1.1×10^{-7}	4.9	3.5
		B	49	6.4×10^{-8}	2.0	5.4
		K	777	3.6×10^{-9}	3.0	1.4

TABLE B-III. - TENSILE TEST RESULTS FOR ALLOY HE42

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	T	1545	1084	20.6	16.4
	T	1509	1055	15.8	14.9
	T	1543	1106	17.3	14.4
	B	1519	1026	27.9	22.4
	K	1451	1154	13.8	14.4
650	T	1321	1032	10.6	12.1
	T	1310	1009	7.7	12.1
	B	1238	948	22.8	24.3
	K	1390	1085	21.5	21.3
760	B	1012	931	15.2	17.8
	K	1111	1052	13.1	14.5

TABLE B-IV. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE42

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	T	165	3.2×10^{-8}	2.2	6.4
		T	154	3.2×10^{-8}	3.8	5.4
		A	531	7.2×10^{-9}	4.1	9.3
		B	453	7.1×10^{-9}	2.7	5.9
		B	826	7.7×10^{-9}	9.3	5.0
		B	692	8.9×10^{-9}	5.9	5.8
		D	535	3.5×10^{-9}	3.4	8.7
		K	2100	1.5×10^{-9}	6.4	4.8
		K	2075	2.8×10^{-9}	4.9	2.9
	900	T	48	7.4×10^{-8}	1.4	4.0
		A	28	3.1×10^{-8}	---	---
		B	93	2.1×10^{-8}	---	---
		B	152	5.8×10^{-8}	7.4	4.7
		D	60	4.3×10^{-8}	0.6	4.5
		K	987	5.7×10^{-9}	3.4	3.0
760	400	T	27	2.2×10^{-7}	0.6	2.0
		A	30	1.1×10^{-7}	2.1	3.0
		B	39	9.7×10^{-8}	4.2	4.0
		B	233	1.1×10^{-8}	1.0	1.0
		D	43	1.1×10^{-7}	3.0	3.0
		K	517	5.0×10^{-9}	3.4	3.0
		K	484	6.0×10^{-9}	2.9	1.0

TABLE B-V. - TENSILE TEST RESULTS FOR ALLOY HE43

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	T	1439	1022	23.5	18.2
	K	1487	1137	18.6	13.2
650	T	1247	948	29.8	26.5
	K	1387	1080	28.9	23.0
760	T	1113	934	13.3	11.8
	K	1100	1038	15.4	12.5

TABLE B-VI. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE43

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	T	778	4.7×10^{-9}	5.9	6.1
		T	722	7.5×10^{-9}	5.9	4.6
		K	2775	1.9×10^{-9}	7.8	13.4
		K	2514	1.8×10^{-9}	5.4	3.3
	900	T	267	2.9×10^{-8}	5.9	2.3
		K	683	1.0×10^{-8}	5.0	5.0
760	400	T	173	1.7×10^{-8}	1.7	1.5
		T	77	4.0×10^{-8}	1.0	3.1
		K	596	5.0×10^{-9}	1.0	3.7

TABLE B-VII. - TENSILE TEST RESULTS FOR ALLOY HE44

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	B	1389	1086	28.5	18.1
	B	1369	947	17.7	20.7
	K	1444	1133	14.9	13.5
650	B	1175	880	29.8	29.6
	K	1337	1043	28.0	22.8
760	B	971	884	14.7	19.1
	K	1037	970	19.0	12.0

TABLE B-VIII. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE44

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	B	388	2.0×10^{-8}	7.4	1.8
		B	395	1.9×10^{-8}	11.6	5.0
		K	1471	2.7×10^{-9}	5.9	5.3
		K	1366	4.3×10^{-9}	4.9	3.4
	900	B	93	9.6×10^{-8}	7.8	4.4
		K	422	1.4×10^{-8}	6.9	4.8
760	400	B	108	2.2×10^{-8}	.5	.7
		K	610	1.3×10^{-9}	0	1.2

TABLE B-IX. - TENSILE TEST RESULTS FOR ALLOY HE45

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	T	1352	975	17.6	18.6
	E	1484	1088	16.7	14.9
650	T	1201	909	23.2	22.2
	E	1339	1040	21.0	19.9
760	T	981	891	8.2	10.7
	E	1086	991	7.3	9.7

TABLE B-X. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE45

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	T	390	9.9×10^{-9}	6.8	6.0
		T	416	9.9×10^{-9}	---	---
		E	1108	2.1×10^{-9}	6.9	3.6
		E	965	3.1×10^{-9}	5.9	2.1
	900	T	73	7.3×10^{-8}	4.9	3.2
		E	274	8.1×10^{-9}	2.0	3.2
760	400	T	98	2.4×10^{-8}	.5	1.7
		E	327	3.0×10^{-9}	1.0	3.2

TABLE B-XI. - TENSILE TEST RESULTS FOR ALLOY HE46

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	B	1304	907	16.4	13.1
	E	1447	1067	17.4	13.4
650	B	1167	977	9.7	13.5
	E	1361	1042	13.0	12.1
760	B	968	893	7.9	9.4
	E	1068	995	4.8	6.9

TABLE B-XII. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE46

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	B	198	3.1×10^{-8}	4.9	3.6
		B	208	1.0×10^{-8}	4.5	2.7
		E	540	3.7×10^{-9}	5.9	4.2
		E	616	2.3×10^{-9}	3.0	3.6
	900	B	90	6.1×10^{-8}	4.9	4.2
		E	152	1.3×10^{-8}	4.5	2.1
760	400	B	141	1.6×10^{-8}	0	2.4
		E	281	8.3×10^{-9}	1.5	2.3

TABLE B-XIII. - TENSILE TEST RESULTS FOR ALLOY HE47

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	B	1301	927	16.5	15.4
	K	1431	1123	12.0	12.3
650	B	1174	880	22.6	17.2
	K	1369	1070	18.2	17.1
760	B	927	869	15.7	12.6
	K	1078	980	7.1	8.8

TABLE B-XIV. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE47

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	B	178	$2.8x10^{-8}$	4.9	4.7
		B	174	$3.1x10^{-8}$	6.4	4.8
		E	459	$7.8x10^{-9}$	6.9	4.8
		E	452	$6.7x10^{-9}$	2.0	1.0
	900	B	45	$2.7x10^{-7}$	9.8	8.7
		E	93	$2.3x10^{-8}$	5.9	2.9
760	400	B	48	$3.3x10^{-8}$	0	.4
		B	48	$5.6x10^{-8}$	1.5	3.7
		E	209	$8.2x10^{-9}$	0	.7
		E	239	$8.6x10^{-9}$	0	1.8

TABLE B-XV. - TENSILE TEST RESULTS FOR ALLOY HE48

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	B	1290	929	5.4	13.1
	K	1429	1091	12.6	14.4
650	B	1173	882	10.4	16.4
	K	1363	1046	15.9	12.6
760	B	920	871	7.5	14.6
	K	934	902	5.6	7.8

TABLE B-XVI. - CREEP RUPTURE TEST RESULTS FOR ALLOY HE48

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	B	162	$2.0x10^{-8}$	5.2	4.5
		B	167	$3.9x10^{-8}$	6.8	4.7
		K	116	$2.5x10^{-8}$	14.7	8.7
		K	410	$8.0x10^{-9}$	4.0	3.7
		K	317	$2.4x10^{-8}$	3.0	1.3
	900	B	12	--------	5.9	1.5
		K	137	$4.3x10^{-8}$	4.5	7.5
760	400	B	50	$2.9x10^{-8}$	2.0	2.1
		K	89	$4.3x10^{-8}$	4.5	7.5

TABLE B-XVII. - AVERAGE PROPERTIES OF ALLOY HE83[a]

(a) Tensile properties

Test temperature, °C	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	1440	967	16.6	21.2
650	1244	914	15.0	17.6

(b) Creep rupture properties

Test temperature, °C	Stress, MPa	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	252	1.0×10^{-8}	3.2	6.8
	900	62	4.3×10^{-8}	1.6	5.4
760	475	60	4.2×10^{-8}	4.1	5.4

[a]Powder metallurgy version of Udimet 700 (ref. 3).

TABLE B-XVIII. - AVERAGE PROPERTIES OF ALLOY HE79[a]

(a) Tensile properties

Test temperature, °C	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	1427	1005	11.1	17.2
650	1227	926	22.6	29.4

(b) Creep rupture properties

Test temperature, °C	Stress, MPa	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	428	1.7×10^{-8}	5.1	8.3
	900	152	8.0×10^{-8}	5.6	13.1
760	475	75	5.5×10^{-8}	2.6	3.0

[a]Variation of Udimet 700 with 8.5 percent cobalt (ref. 3).

TABLE B-XIX. - AVERAGE PROPERTIES OF ALLOY HE74[a]

(a) Tensile properties

Test temperature, °C	Heat treat type	Ultimate strength, MPa	Yield strength, MPa	Elongation, percent	Reduction of area, percent
25	A	1395	962	11.7	14.9
	B	1405	985	19.6	16.3
	G	1405	973	19.6	16.4
650	A	1197	877	12.8	18.3
	B	1240	900	21.7	13.6
	G	1208	884	20.1	10.6

(b) Creep rupture properties

Test temperature, °C	Stress, MPa	Heat treat type	Life, hr	Minimum creep rate, sec^{-1}	Elongation, percent	Reduction of area, percent
650	825	A	222	4.3×10^{-8}	6.9	9.3
		B	267	2.7×10^{-8}	3.8	14.8
		G	203	3.8×10^{-8}	5.1	9.3
	900	A	74	1.6×10^{-7}	3.8	10.7
		B	87	1.2×10^{-7}	19.6	16.3
		G	62	2.0×10^{-7}	6.7	11.6
760	475	A	51	9.2×10^{-8}	1.1	3.7
		B	46	9.5×10^{-8}	4.9	3.9
		G	43	8.4×10^{-8}	2.5	3.0

[a]Cobalt free version of Udimet 700 alloy (ref. 4).

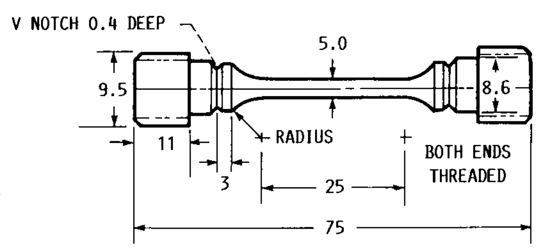

FIGURE 1. - SKETCH OF TEST SPECIMEN. ALL DIMENSIONS IN MILLIMETERS.

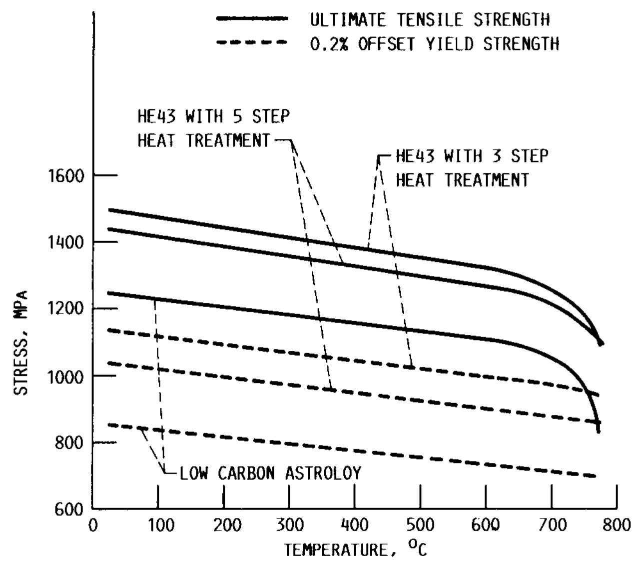

FIGURE 2. - TENSILE PROPERTIES OF ALLOY HE43 WITH 3- AND 5- STEP HEAT TREATMENT. COMPARISON IS MADE TO LOW CARBON ASTROLOY, A POWDER METALLURGY VERSION OF UDIMET 700[7].

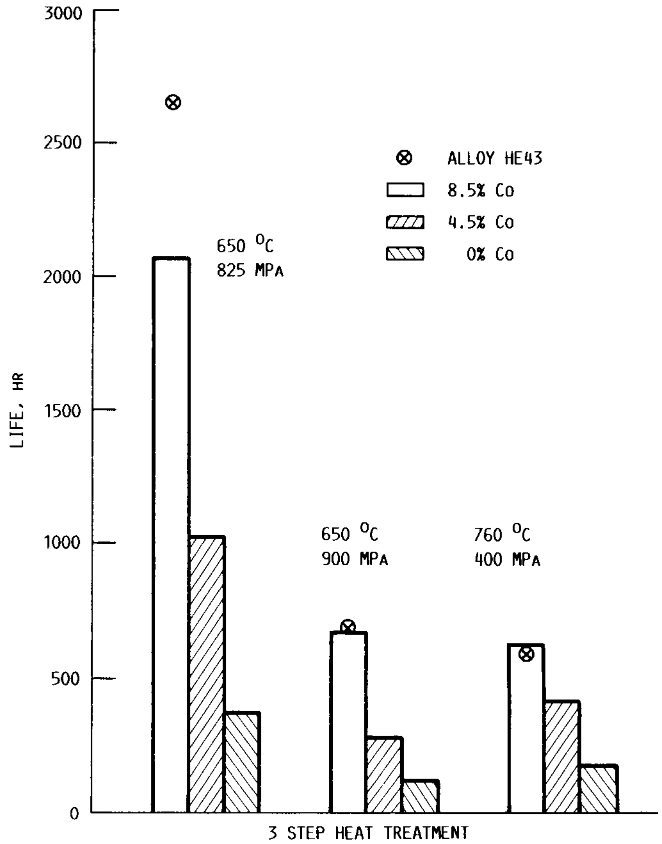

FIGURE 3. - EFFECT OF COBALT CONTENT ON CREEP RUPTURE LIVES OF ALLOYS WITH A THREE-STEP HEAT TREATMENT UNDER THREE TEST CONDITIONS: LIVES ARE AVERAGE OF HE41, HE42 AND HE43 FOR 8.5% COBALT; HE44, HE45, HE46 FOR 4.5% COBALT; HE47 AND HE48 FOR 0% COBALT. CIRCLE INDICATES LIVES OF ALLOY HE43.

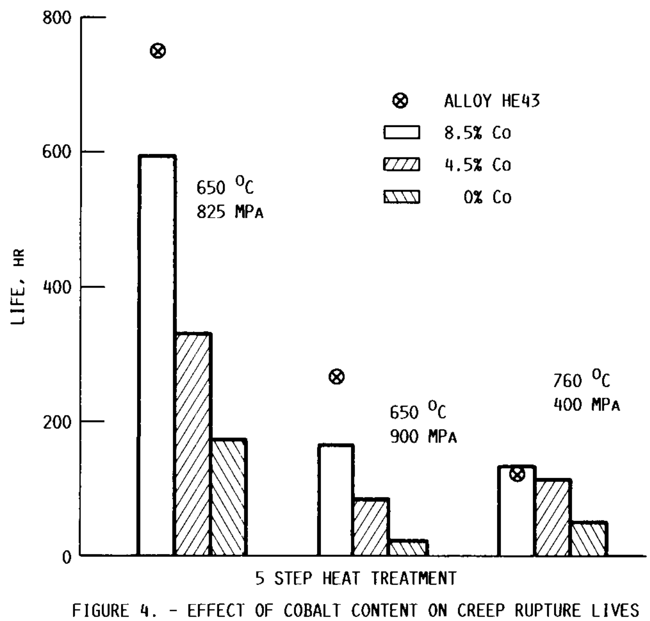

FIGURE 4. - EFFECT OF COBALT CONTENT ON CREEP RUPTURE LIVES OF ALLOYS WITH A FIVE-STEP HEAT TREATMENT UNDER THREE TEST CONDITIONS: LIVES ARE AVERAGE OF HE41, HE42 AND HE43 FOR 8.5% COBALT; HE44, HE45, HE46 FOR 4.5% COBALT; HE47 AND HE48 FOR 0%. CIRCLE INDICATES LIVES OF ALLOY HE43.

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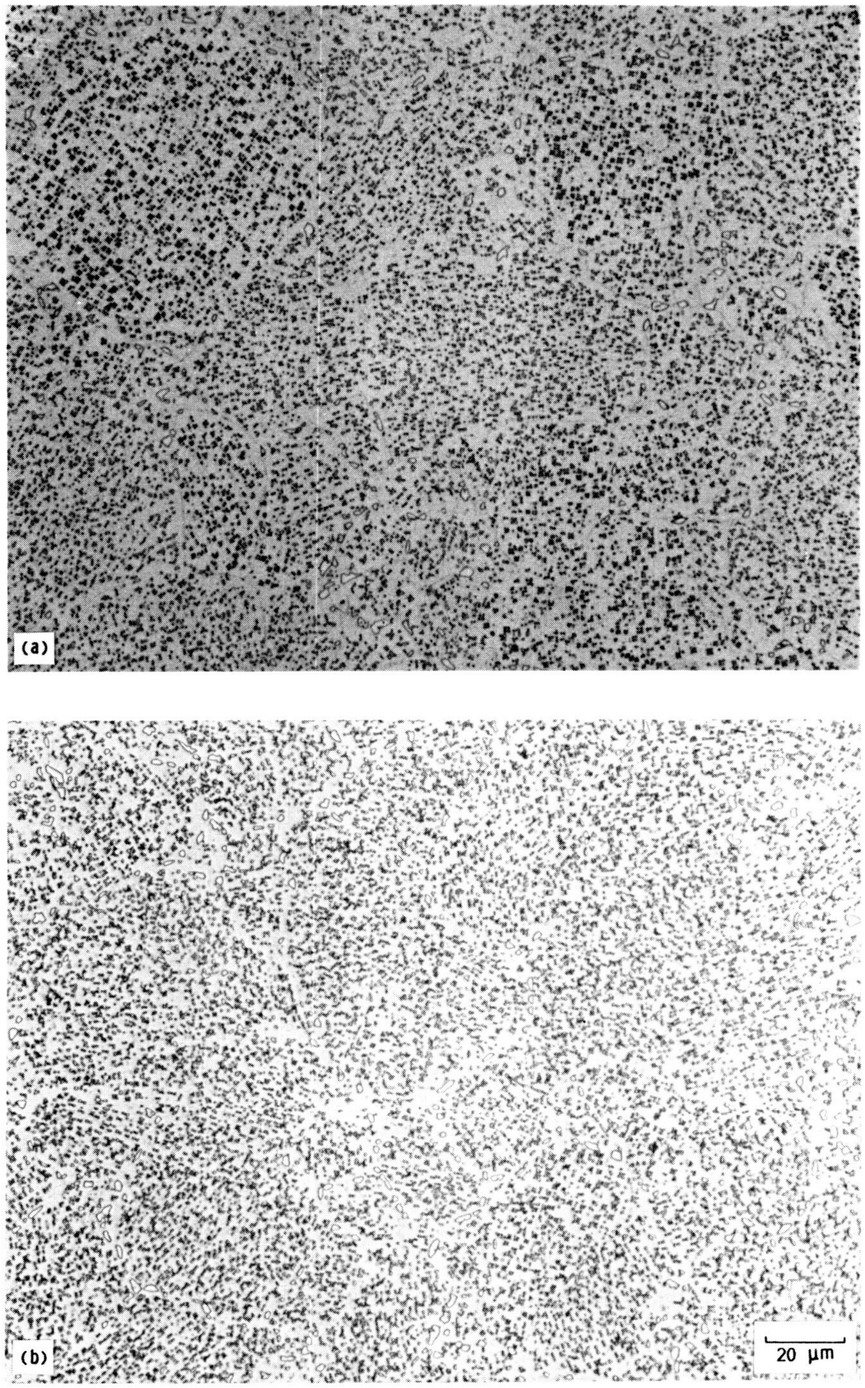

FIGURE 5. - ALLOY HE43 SHOWING TYPICAL MICROSTRUCTURE FOR A (A) 3-STEP, AND (B) 5-STEP HEAT TREATMENT.

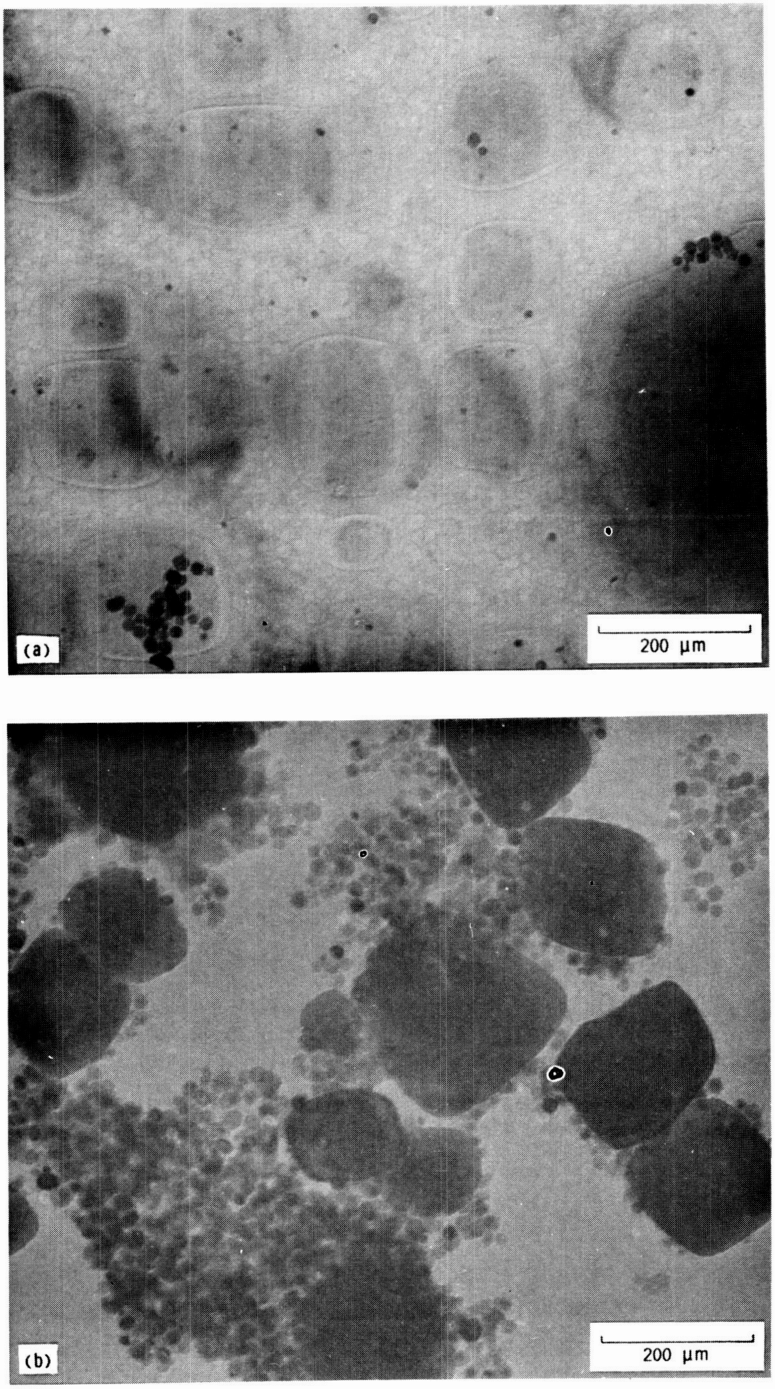

FIGURE 6. - TRANSMISSION ELECTRONMICROGRAPHS OF ALLOY HE41 WITH 5-STEP TREATMENT T: (A) THIN FILM SPECIMEN, (B) EXTRACTED PARTICLES.

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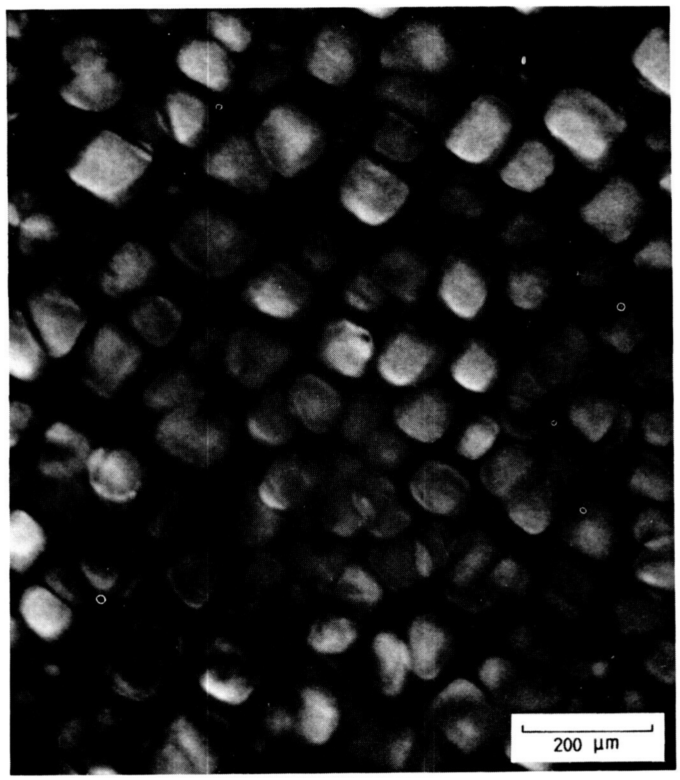

FIGURE 7. - TRANSMISSION ELECTRON MICROGRAPH OF ALLOY HE41 WITH
3-STEP HEAT TREATMENT K.

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15. Supplementary Notes

16. Abstract

Eight superalloys derived from Udimet 700 were prepared by powder metallurgy, hot isostatically pressed, heat treated and their tensile and creep rupture properties determined. Several of these alloys displayed properties superior to those of Udimet 700 similarly prepared, in one case exceeding the creep rupture life tenfold. Filter clogging by extracted gamma prime, its measurement and significance are discussed in an appendix.

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